Practitioner's Docket

MSU 4.1-572

PATENT

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IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Jes Asmussen and Wen-Shin Huang

Application No.: 10 / 073,710 Group No.: 1762

Filed: February 11, 2002 Examiner: Eric B. Fuller

or: PROCESS FOR SYNTHESIZING UNIFORM NANOCRYSTALLINE FILMS

Mail Stop Appeal Brief—Patents Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

TRANSMITTAL OF APPEAL BRIEF (PATENT APPLICATION—37 C.F.R. § 1.192)

NOTE: The phrase "the date on which" an "appeal was taken" in 35 U.S.C. 154(b)(1)(A)(ii) (which provides an adjustment of patent term if there is a delay on the part of the Office to respond within 4 months after an "appeal was taken") means the date on which an appeal brief under § 1.192 (and not a notice of appeal) was filed. Compliance with § 1.192 requires that: 1. the appeal brief fee (§ 1.17(c)) be paid (§ 1.192(a)); and 2.the appeal brief complies with § 1.192(c)(1) through (c)(9). See Notice of September 18, 2000, 65 Fed. Reg. 56366, 56385-56387 (Comment 38).

1. Transmitted herewith, in triplicate, is the APPEAL BRIEF in this application, with respect to the Notice of Appeal filed on $\frac{01/18/05}{}$

NOTE: "Appellant must, within two months from the date of the notice of appeal under § 1.191 or within the time allowed for reply to the action from which the appeal was taken, if such time is later, file a brief in triplicate. . . " 37 C.F.R. § 1.192(a) (emphasis added).

CERTIFICATION UNDER 37 C.F.R. §§ 1.8(a) and 1.10*

(When using Express Mail, the Express Mail label number is mandatory; Express Mail certification is optional.)

I hereby certify that, on the date shown below, this correspondence is being:

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(Transmittal of Appeal Brief [9-6.1]—page 1 of 4)

^{*} Only the date of filing (§ 1.6) will be the date used in a patent term adjustment calculation, although the date on any certificate of mailing or transmission under § 1.8 continues to be taken into account in determining timeliness. See § 1.703(f). Consider "Express Mail Post Office to Addressee" (§ 1.10) or facsimile transmission (§ 1.6(d)) for the reply to be accorded the earliest possible filing date for patent term adjustment calculations.

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2. STA	TUS OF APPLICAN	r					
This a	This application is on behalf of						
	other than a sma	III entity.					
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	A statement:						
·	☐ is attached.						
	🖾 was already	filed.					
3. FEE	FOR FILING APPEA	AL BRIEF					
Pursu	ant to 37 C.F.R. § 4	11.20(b)(2), the fee fo	r filing the Ap	peal Brief is:			
×	small entity		\$170	.00 -\$250.00			
<u> </u>	other than a sma	all entity	\$340	.00- \$500 . 00			
•	·						
Appeal Brief fee due \$ 250.00							
	ENSION OF TERM						
NOTE: 37 C.F.R. § 1.704(b) " an applicant shall be deemed to have failed to engage in reasonable efforts to conclude processing or examination of an application for the cumulative total of any periods of time in excess of three months that are taken to reply to any notice or action by the Office making any rejection, objection, argument, or other request, measuring such three-month period from the date the notice or action was mailed or given to the applicant, in which case the period of adjustment set forth in § 1.703 shall be reduced by the number of days, if any, beginning on the day after the date that is three months after the date of mailing or transmission of the Office communication notifying the applicant of the rejection, objection, argument, or other request and ending on the date the reply was filed. The period, or shortened statutory period, for reply that is set in the Office action or notice has no effect on the three-month period set forth in this paragraph."							
NOTE:	NOTE: The time periods set forth in 37 C.F.R. § 1.192(a) are subject to the provision of § 1.136 for patent applications. 37 C.F.R. § 1.191(d). See also Notice of November 5, 1985 (1060 O.G. 27).						
NOTE:	NOTE: As the two-month period set in § 1.192(a) for filing an appeal brief is not subject to the six-month maximum period specified in 35 U.S.C. § 133, the period for filing an appeal brief may be extended up to seven months. 62 Fed. Reg. 53,131, at 53,156; 1203 O.G. 63, at 84 (Oct. 10, 1997).						
The proceedings herein are for a patent application and the provisions of 37 C.F.R. § 1.136 apply.							
	(complete (a) or (b), as applicable)						
(a) Applicant petitions for an extension of time under 37 C.F.R. § 1.136 (fees: 37 C.F.R. § 1.17(a)(1)-(5)) for the total number of months checked below:							
	Extension	Fee for other th		ee for			
_ '	(months)	small entity		all entity			
	one month	\$ 110.00 \$ 430.00	The second secon	55.00 215.00			
	two months three months	\$ 430.00		490.00	•		
	four months	\$ 1,530.00	· · · · · · · · · · · · · · · · · · ·	765.00			
	five months	\$ 2,080.00	•	,040.00			
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		(check and complete the next item, if applicable)
	į	An extension for months has already been secured, and the fee paid therefor of \$ is deducted from the total fee due for the total months of extension now requested.
		Extension fee due with this request \$
•		or .
(b) [2	t	Applicant believes that no extension of term is required. However, this conditional petition is being made to provide for the possibility that applicant has nadvertently overlooked the need for a petition and fee for extension of time.
5. TO	ΓAL	FEE DUE
The t	otal	fee due is:
	A	Appeal brief fee \$ 250.00
	Ε	Extension fee (if any) \$ _ 0 . 0 0
		TOTAL FEE DUE \$
6. FEE	E PA	YMENT
⊠ A	\ttac	hed is a ⊠ check ☐ money order in the amount of \$ 250.00
	utho	orization is hereby made to charge the amount of \$
		o Deposit Account No
		o Credit card as shown on the attached credit card information authorization orm PTO-2038.
WARNI	NG:	Credit card information should not be included on this form as it may become public.
	-	ge any additional fees required by this paper or credit any overpayment in the ler authorized above.
Α	du	olicate of this paper is attached.
7. FEE	DE	FICIENCY
NOTE:	nece six-r abar ence to ac	ere is a fee deficiency and there is no authorization to charge an account, additional fees are essary to cover the additional time consumed in making up the original deficiency. If the maximum month period has expired before the deficiency is noted and corrected, the application is held addened. In those instances where authorization to charge is included, processing delays are puntered in returning the papers to the PTO Finance Branch in order to apply these charges prior ction on the cases. Authorization to change the deposit account for any fee deficiency should be cked. See the Notice of April 7, 1986, 1065 O.G. 31-33.
∑ If	any	additional extension and/or fee is required,
		AND/OR
⊠ If	any	additional fee for claims is required, charge:
X) D	Deposit Account No. 13-0610
. =		Credit card as shown on the attached credit card information authorization form
WARNII	VG:	Credit card information should not be included on this form as it may become public.
		(Transmittal of Appeal Brief [9-6.1]—page 3 of 4)

If an additional extension of time is required, please consider this a petition therefor.

Date: 03/10/05

Reg. No.: 20,931

Customer No.: 21036

SIGNATURE OF PRACTITIONER

Ian C. McLeod

(type or print name of practitioner)

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(Transmittal of Appeal Brief [9-6.1]—page 4 of 4)

Attorney Docket No. MSU 4.1-572

Appl. No. 10/073,710

Appeal Brief Dated: March 10, 2005

Appeal Brief

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appln. No. : 10/073,710 Confirmation No. 5422

Applicants : Jes Asmussen and Wen-Shin Huang

Filed : February 11, 2002

Title : PROCESS FOR SYNTHESIZING UNIFORM

NANOCRYSTALLINE FILMS

TC/A.U. : 1762

Examiner : Eric B. Fuller

Docket No. : MSU 4.1-572

Customer No.: 21036

MAIL STOP APPEAL BRIEF - Patents COMMISSIONER FOR PATENTS P. O. BOX 1450 ALEXANDRIA VA 22313-1450

APPEAL BRIEF UNDER 37 C.F.R. § 41.37 Under Part 41 subpart B

Sir:

This is an appeal from a final rejection in the above entitled application. The claims on appeal are set forth as Claims Appendix A. An oral hearing will be requested. Enclosed is the fee due upon filing of the Brief.

03/15/2005 HALI11 00000017 10073710

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(I) Real Party in Interest

The real party in interest is Michigan State University, East Lansing, Michigan.

(ii) Related Appeals and Interferences

There are no related appeals or interferences.

(iii) Status of Claims

Claims 1 to 5 and 8 to 19 are on appeal.

Claims 6 and 7 were subject to a restriction requirement and were non-elected without traverse.

Claim 19 was added during the prosecution of the application in the Amendment filed August 9, 2004.

(iv) Status of Amendments

An amendment under 37 CFR 1.116(b) was filed to correct antecedent basis problems in Claim 1 (Appendix B).

(v) Summary of Claimed Subject Matter

Claim 1 is the only independent claim. The claim generally relates to a process wherein a chamber in which a plasma 56 is generated in a chamber 40 to deposit diamond as shown in Figures 1 to 1E. The

novelty in Claim 1 particularly relates to part (c) The plasma 56 used is lines 36 to 46. specifically set forth in Claim 1, which is 90% or more by volume argon along with methane and optionally hydrogen at a pressure of between 50 and 300 Torr. plasma 56 is essentially free of oxygen or nitrogen and the chamber 40 is free from leaks of nitrogen or oxygen. This is disclosed on page 3, lines 5 to 17 of the specification. The result is the ability to generate nanocrystalline uniform diamond films with a diameter greater than about 8 cm (see Claim 14) and a surface area greater than about 20 cm2 (Claim 2).

The preferred prior art reactor is shown in Figures 1 to 1F and is described in the specification at pages 12 to 19 of the specification. The essential elements are set forth hereinafter in Claim 1.

-1-

A process for depositing a nanocrystalline diamond film with a grain size between 1 and 100 nm on a surface of a substrate, which comprises:

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providing a plasma (56) generating (a) apparatus (10) for depositing the diamond film on the substrate 50) from the plasma (56), including a plasma source employing a radiofrequency, including UHF or microwave, wave coupler means which is metallic and in the shape of a hollow cavity 12) and which is excited in a TM mode of resonance, and wherein an insulated the chamber means (40) has a central longitudinal axis (A-A) in common with the coupler means and is mounted in closely spaced and sealed relationship to an area of the coupler means with an opening from the chamber means at one end; gas supply means (36) for providing a gas which is ionized to form the plasma (56) in the chamber means (40), wherein the radiofrequency wave applied to the coupler means creates and maintains the plasma around the central longitudinal axis (A-A) in the chamber means (40); movable metal plate means (16) in the coupler cavity mounted means perpendicular to the central longitudinal axis (A-A) and movable along the central longitudinal axis (A-A) towards and away from the chamber means (40); and a

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- movable probe means 30) connected to and extending inside the coupler means for coupling the radiofrequency waves to the coupler means;
 - (b) providing the substrate <u>(50)</u>, wherein the surface to be placed in the plasma has been roughened and cleaned; and
 - providing the substrate (50) in the insulated chamber means on a substrate holder adjacent to the plasma generated in the chamber means (40), wherein the gas in the chamber means (40) is at a pressure between 50 and 300 Torr in the presence of the radiofrequency waves for generating the plasma (56), wherein the gas is ninety percent by volume or more of argon along with methane and optionally hydrogen and essentially free from oxygen or nitrogen and wherein the chamber means (40) is essentially free from leaks of nitrogen or oxygen or mixtures thereof into the chamber means, so as to generate the plasma and to the nanocrystalline diamond film the deposit substrate.

(3) Claims 8, 10, 12 and 15 are separately argued for purposes of patentability in that provide the reaction conditions for producing thick least about 50 diamond (at nanocrystalline disclosed on page 6, third micrometers) as paragraph of the specification and as set forth in Claim 5 and the large surface area or diameter of the diamond as in Claims 2 and 14, as set forth in the third paragraph and fourth paragraph leading to page 7. Claim 8 calls for the "thermal floating" as set forth on page 5 lines 19 to 29 of the specification. 10 calls for the specific preferred pressures as set forth on page 5, line 27 to page 6, line 2 of the Claim 12 calls for less than 10 ppm of specification. oxygen or nitrogen as set forth on page 6, second full paragraph of the specification. Claim 15 calls for about 1% methane as the carbon material for forming the diamond as set forth on page 3, lines 6 to 10 of the specification.

(vi) Grounds of Rejection to Be Reviewed on Appeal

Claims 1-5, 8-12, 14-17, and 19 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S. 4,961,958) in further view of Asmussen et al. (U.S. 5,311,103).

Claims 13 and 18 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S. 4,961,958), in further view of Asmussen et al. (U.S. 5,311,103), as applied to Claims 1 and 2 above, and further in view of Herb et al. (U.S. 5,273,790).

Claims 1-5, 8-12, 14-17, and 19 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S. 4,961,958), in further view of Asmussen et al. (U.S. 4,585,668).

Claims 13 and 18 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S. 4,961,958), in further view of Asmussen et al. (U.S.

4,585,668), as applied to Claims 1 and 2 above, and further in view of Herb et al. (U.S. 5,273,790).

Claims 1-5, 8-12, 14-17, and 19 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S. 4,961,958), in further view of Asmussen et al. (U.S. 4,906,900).

Claims 13 and 18 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S. 4,961,958), in further view of Asmussen et al. (U.S. 4,906,900), as applied to Claims 1 and 2 above, and further in view of Herb et al. (U.S. 5,273,790).

Claims 1-5, 8-12, 14-17, and 19 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S. 4,961,958), in further view of Asmussen et al. (U.S. 4,727,293).

Claims 13 and 18 were rejected under 35 U.S.C. 103(a) as being unpatentable over Janesen et al. (U.S. 4,925,701), in view of Desphandey et al. (U.S.

4,961,958), in further view of Asmussen et al. (U.S. 4,727,293), as applied to Claims 1 and 2 above, and further in view of Herb et al. (U.S. 5,273,790).

Claims 1-5, 8-12, 14-17, and 19 were rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over Claims 1, 2 and 4 of U.S. Patent No. 4,585,668 in view of Janesen et al. (U.S. 4,925,701) in view of Desphandey et al. (U.S. 4,961,958).

Claims 1-5, 8-12, 14-17, and 19 were rejected under the judicially created doctrine of obviousness-type double patenting as being unpatentable over Claims 22-27 of U.S. Patent No. 4,585,668 in view of Janesen et al. (U.S. 4,925,701) in view of Desphandey et al. (U.S. 4,961,958).

The Applicants believe that these rejections are incorrect for the reasons set forth hereinafter.

(vi) Argument

(1) Claims 1 to 5, 8 to 12 and 14 to 17 were rejected under 35 USC 103(a) as being unpatentable over Janesen et al (U.S. Patent No. 4,925,701), in view of

Desphandey et al (U.S. patent No. 4,961,958), in further view of <u>Asmussen</u> et al (U.S. patent No. 5,311,103). This rejection is traversed.

The present invention resides in the discovery that in order to prepare nanocrystalline diamond in the type of reactor specified in the preamble, the conditions set forth in independent Claim 1 must be met. None of the references suggest a plasma and chamber for generating the nanocrystalline diamond which must be (1) essentially free from oxygen or nitrogen in the plasma, and (2) without leaking nitrogen or oxygen into the chamber of the apparatus under these conditions.

Janesen et al (U.S. Patent No. 4,925,701) does not describe the preparation of nanocrystalline diamond. The reactor described is not of the type used in Applicants' process and uses heated filaments and the metal from the heated filaments are well known to contaminate the diamond films. Janesen describes the conventional CVD deposition chemistry that produces polycrystalline diamond films. The gases used were

hydrogen and a carbon source, rather than a 90% or more argon plasma as set forth in Claim 1. About the only part of the reference which is relevant is roughening the surface which is an old step. Column 11, lines 32 to 55 of Janesen et al describe the polycrystalline film properties and has nothing to do with processing of the diamond and are related to testing the film produced from the process. The reason for keeping the gaseous impurities low in the Applicants' process is to maintain the nanocrystalline nature of of impurities The presence allows the film. polycrystalline film growth.

Desphandey et al (U.S. Patent No. 4,961,958) describes a chamber which uses a heated filament 46 which is a different type reactor apparatus not claimed by Applicants. Again metal from such filaments are well known to contaminate the diamond film. Carbon is evaporated in the presence of hydrogen for deposit of diamond on a substrate. Column 5, lines 30 to 42 discloses "enhancing" the deposition of diamond with argon. Here it is important to note that Desphandey's

deposition takes place in a completely different process region, i.e. 1m Torr - 20m Torr. This is orders of magnitude below the Applicants' several deposition parameter of 50-300 Torr. Thus the plasma chemistry of <u>Desphandey</u> is completely different. plasma is a cold discharge at these pressures versus in the Applicants' process discharge that is in a thermal The deposition chemistry is thus very environment. different. The reason for the introduction of Argon in the Applicant's reaction is to control the chemistry and thereby produce only nanocrystalline diamond films. Nothing approaching the use of 90% or more of argon with methane and the optional use of hydrogen as in the claims is suggested in this reference.

Asmussen et al ('103) describes the type of electrodeless plasma reactor encompassed by the claims; however, there is no disclosure of the process claimed as discussed above. Thus the combination of references would not possibly produce the claimed process. Reconsideration of this rejection is requested.

Claims 13 and 18 were rejected under 35 USC 103(a) as unpatentable over previously applied references in view of <u>Herb</u> et al (U.S. Patent No. 5,273,790). Molybdenum is well known for use as a substrate holder. The patentability of these claims depends upon the independent claims upon which these claims depend. The specific limitations of Claims 13 and 18 are merely preferred. Reconsideration of this rejection is requested.

Claims 1-5, 8-12, 14 to 17 were rejected over Janesen et al (US 4,925,701), in view of Desphandey et al (US 4,961,958), in further view of Asmussen et al Asmussen et al (U.S. Patent 4,585,668) (U.S. `668). describes a reactor of the type described in Asmussen invention is not in the ('103). The applicants' specific type of reactor apparatus without electrodes, but in a different deposition process. The deposition is completely different in the chemistry The arguments previously presented improved process. apply equally to this combination of references. Reconsideration of this rejection is requested.

Claims 13 to 18 were rejected under 35 USC 103(a) over <u>Janesen</u> et al (US 4,925,701), in view of <u>Desphandey</u> et al (US 4,961,958), in further view of <u>Asmussen</u> et al (US 4,585,668), and further in view of <u>Herb</u> et al (US 5,273,790). Silicon is a well known substrate for diamond deposition and molybdenum is a well known substrate holder. These materials are merely preferred. The points as to Claims 13 and 18 are set forth above apply to this rejection as well. Reconsideration is requested.

Claims 1 to 5, 8 to 12 and 14 to 17 were rejected over Janesen et al (US 4,925,701), in view of Desphandey et al (US 4,961,958), in further view of <u>Asmussen</u> et al (US 4,906,900). The <u>Asmussen</u> et al ('900) reference merely describes another type of electrodeless reactor of the type used in the claimed The disclosures of Janesen al and et process. Desphandey have been discussed above. The combination of references does not teach the claimed invention. Reconsideration is requested.

Claims 13 and 18 were rejected under 35 USC 103(a) as being unpatentable over <u>Janesen</u> et al (US 4,925,701), in view of <u>Desphandey</u> et al (US 4,961,958), in further view of <u>Asmussen</u> et al (US 4,906,900), as applied to claims 1 and 2 above, and further in view of <u>Herb</u> et al (US 5,273,790). The points made as to these Claims 13 and 18 are set forth above and apply to this rejection as well. Reconsideration is requested.

Claims 1-5, 8-12 and 14-17 were rejected over <u>Janesen</u> et al (US 4,925,701), in view of <u>Desphandey</u> et al (US 4,961,958), in further view of <u>Asmussen</u> et al (US 4,727,293). <u>Asmussen</u> '293 describes the type of reactor used by Applicants. <u>Janesen</u> et al and <u>Desphandey</u> et al have been discussed previously and combined with <u>Asmussen</u> et al ('293) could not possibly produce the claimed invention.

Claim 13 and 18 were rejected under 35 USC 103(a) over <u>Janesen</u> et al (US 4,925,701), in view of <u>Desphandey</u> et al (US 4,961,958), in further view of <u>Asmussen</u> et al (US 4,727,293), as applied to Claims 1 and 2 above, and further in view of <u>Herb</u> et al (US

5,273,790). Asmussen '293 describes another preferred electrodeless reactor. The points previously made as to Claims 13 and 18 apply to this rejection as well. Reconsideration is requested.

using hindsight in rejection is attempt to reconstruct the claimed invention, which is The reactor used in Applicants' process is a process. known as described in the Asmussen et al patents. The need for strict removal of nitrogen and oxygen from the plasma was not recognized by Asmussen et al or any of Janesen et al and Desphandey et the other references. primary references, describe which the al, are electrode type reactors which are not suitable for the In fact they describe a completely present process. different deposition process. The deposition processes and chemistries do not produce nanocrystalline diamond. None of the references alone or in combination set forth any suggestion of the claimed invention. skilled in the art could not have derived the claimed invention from the combinations of references cited.

- (2) Claim 8 is separately patentable. Claim 8 calls for a floating substrate temperature which is allowed to float between 575°C and 900°C on a side of the substrate exposed to the plasma. There is no suggestion in any of the references alone or in combination of using this procedure to grow the diamond film under the conditions set forth in Claim 8. This is the procedure of the Example in the specification (page 25, line 5).
- (3) Claim 10 is separately patentable. the references describe or suggest alone or combination the process with the specific reaction conditions of this claim. Ιt is these reaction which specifically produce the conditions nanocrystalline diamond of Claims 2, 5 and 14. The combination of references would not suggest reaction conditions of Claim 10 coupled with Claim 1. Reconsideration is requested.
- (4) Claim 12 specifically sets forth the amount of oxygen and/or nitrogen to be less than 10 ppm, as disclosed on page 6, second full paragraph, of

the specification. Again there is no suggestion of these preferred reaction conditions of Claim 12 which produces the nanocrystalline diamond of Claims 2, 5 and 14. Reconsideration is requested.

(5) Claim 13 calls for 1% methane which is the carbon containing gas which produces the diamond as disclosed on page 26, last paragraph, of the specification. None of the references alone or in combination suggest this preferred reaction condition.

Claims 1 to 5, 8 to 12, 14 to 17 and 19 were rejected based upon double patenting. As can be seen al and previous arguments, <u>Janesen</u> et from the Desphandey et al use electrode type reactors which are suitable for Applicants' process and are not invention is claimed by Applicants. The present earlier Asmussen patents. on the improvement an Reconsideration of this rejection is requested.

Conclusion

It is believed that the rejection of Claims 1 to 5 and 8 to 19 should be reversed. Reversal of the rejections is respectfully requested.

Respectfully,

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Attachments - Appendix A - Claims -Appendix B - Amendment Under 37 CFR 1.116(b)

CLAIMS APPENDIX A

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-1-

A process for depositing a nanocrystalline diamond film with a grain size between 1 and 100 nm on a surface of a substrate, which comprises:

(a) providing a plasma generating apparatus for depositing the diamond film on the substrate from plasma including a plasma source employing a the radiofrequency, including UHF or microwave, coupler means which is metallic and in the shape of a hollow cavity and which is excited in a TM mode of resonance, and wherein the chamber means has a central longitudinal axis in common with the coupler means and is mounted in closely spaced and sealed relationship to an area of the coupler means with an opening from the supply means end; gas chamber means one at providing a gas which is ionized to form the plasma in the chamber means, wherein the radiofrequency wave applied to the coupler means creates and maintains the plasma around the central longitudinal axis in the chamber means; movable metal plate means in the cavity

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- mounted in the coupler means perpendicular to the central longitudinal axis and movable along the central longitudinal axis towards and away from the chamber means; and a movable probe means connected to and extending inside the coupler means for coupling the radiofrequency waves to the coupler means;
 - (b) providing the substrate, wherein the surface to be placed in the plasma has been roughened and cleaned; and
 - (c) providing the substrate in the insulated chamber on a substrate holder adjacent to the plasma generated in the chamber, wherein the gas in chamber is at a pressure between 50 and 300 Torr in the presence of the radiofrequency waves for generating the plasma, wherein the gas is ninety percent by volume or along with methane and optionally argon more of hydrogen and essentially free from oxygen or nitrogen and wherein the chamber is essentially free from leaks of nitrogen or oxygen or mixtures thereof into the chamber, so as to generate the plasma and to deposit the nanocrystalline diamond film on the substrate.

-2-

The process of Claim 1 wherein the substrate has a dimension with a surface area greater than about $20~{\rm cm}^2.$

-3-

The process of Claim 1 or 2 wherein the microwave is at 2.45 GHz.

-4-

The process of Claim 1 or 2 wherein the microwave is at 915 MHz.

-5-

The process of Claim 1 or 2 wherein the film has a thickness of at least about 50 nm micrometers.

-8-

The process of Claim 1 wherein the substrate is allowed to thermally float at a temperature between about 575°C and 900°C on a side exposed to the plasma.

-9-

The process of Claim 1 wherein diamond particles are used for providing the roughened surface by abrasion and wherein the diamond particles have a grain size between about 0.1 to several micrometers, which surface is then cleaned.

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The process of Claim 1 wherein the pressure on the gas is between about 60 and 240 Torr and at a flow rate of between about 50 and 200 sccm.

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-11-

The process of Claims 1 or 2 wherein the probe means is elongate and is mounted in the coupler along the central longitudinal axis of means chamber means and coupler means with an end of probe means in spaced relationship to the chamber means; and wherein stage means in the opening of the chamber which forms part of the cavity and provides for mounting the substrate, the stage means having a support surface which is in a plane around the longitudinal axis and which is pre-adjusted towards and away from the plasma in the chamber means so that the substrate can be coated with the diamond film from the plasma.

-12-

The process of Claims 1 or 2 wherein the insulated chamber is evacuated so that there is less than about 10 ppm of combined oxygen and nitrogen or nitrogen or oxygen alone as the gas which generates the plasma is provided in the chamber.

-13-

The process of Claims 1 or 2 wherein the substrate is silicon and wherein the substrate holder is molybdenum.

-14-

The process of any one of Claims 1 or 2 wherein the substrate on which the diamond is deposited has a surface area with a diameter which is greater than about 8 cm.

-15-

The process of Claim 1 wherein the gas contains about 1% methane.

-16-

The process of Claim 1 wherein the mode of the plasma is selected from the group consisting of TM012 and TM013.

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-17-

The process of Claim 1 wherein at pressures of greater than about 250 Torr the stage means can be optionally cooled.

-18-

The process of Claim 1 wherein the substrate is a silicon carbide seal and the holder is molybdenum which shields a first portion of the seal while allowing a portion of the seal to be coated with the nanocrystalline diamond.

-19-

The process of Claim 1 wherein the apparatus has a static magnetic field around the plasma which aids in coupling radiofrequency energy at electron cyclotron resonance and aids in confining ions in the plasma in an electrically insulated chamber means in the coupler means.

APPENDIX B

Attorney Docket No. MSU 4.1-572 Appl. No. 10/073,710 Amdt. Dated: February 21, 2005 Reply to Office Action of 10/29/2004



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appln. No. : 10/073,710 Confirmation No. 5422

Applicants : Jes Asmussen and Wen-Shin Huang

Filed : February 11, 2002

Title : PROCESS FOR SYNTHESIZING UNIFORM

NANOCRYSTALLINE FILMS

TC/A.U. : 1762

Examiner : Eric B. Fuller

Docket No. : MSU 4.1-572

Customer No.: 21036

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AMENDMENT UNDER 37 CFR 1.116(b)

Sir:

In response to the Office Action mailed October 29, 2004, the Applicants amend and remark as follows:

Amendments to the Claims are reflected in the Listing of Claims which begins on page 2 of this paper.

Remarks begin on page 10 of this paper.

AMENDMENTS TO THE CLAIMS

This listing of claims will replace all prior versions, and listings, of claims in the application:

LISTING OF CLAIMS

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-1-(Currently Amended)

A process for depositing a nanocrystalline diamond film with a grain size between 1 and 100 nm on a surface of a substrate, which comprises:

(a) providing a plasma generating apparatus for depositing the diamond film on the substrate from the plasma, including a plasma source employing a radiofrequency, including microwave, UHF or coupler means which is metallic and in the shape of a hollow cavity and which is excited in a TM mode of resonance, and wherein an insulated the chamber means has a central longitudinal axis in common with the coupler means and is mounted in closely spaced and sealed relationship to an area of the coupler means with an opening from the chamber means at one end; gas supply means for providing a gas which is ionized to form the plasma in the chamber means, wherein the

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radiofrequency wave applied to the coupler creates and maintains the plasma around the central longitudinal axis (A-A) in the chamber means; movable metal plate means in the cavity mounted in the coupler 20 . means perpendicular to the central longitudinal axis and movable along the central longitudinal axis towards and away from the chamber means; and a movable probe means connected to and extending inside the coupler means for coupling the radiofrequency waves to the coupler means;

- providing the substrate, wherein the surface to be placed in the plasma has been roughened and cleaned; and
- 30 (c) providing the substrate in the insulated chamber means on a substrate holder adjacent to the plasma generated in the chamber means, wherein the gas in the chamber means is at a pressure between 50 and 300 Torr in the presence of the radiofrequency waves 35 for generating the plasma, wherein the gas is ninety percent by volume or more of argon along with methane and optionally hydrogen and essentially free

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oxygen or nitrogen and wherein the chamber <u>means</u> is essentially free from leaks of nitrogen or oxygen or mixtures thereof into the chamber <u>means</u>, so as to generate the plasma and to deposit the nanocrystalline diamond film on the substrate.

-2-(Original)

The process of Claim 1 wherein the substrate has a dimension with a surface area greater than about $20~{\rm cm}^2.$

-3-(Original)

The process of Claim 1 or 2 wherein the microwave is at 2.45 GHz.

-4-(Original)

The process of Claim 1 or 2 wherein the microwave is at 915 MHz.

-5-(Original)

The process of Claim 1 or 2 wherein the film has a thickness of at least about 50 nm micrometers.

-6-(Withdrawn)

A nanocrystalline film prepared by the process of Claim 1.

-7-(Withdrawn)

A nanocrystalline film prepared by the process of Claim 2.

-8-(Original)

The process of Claim 1 wherein the substrate is allowed to thermally float at a temperature between about 575°C and 900°C on a side exposed to the plasma.

-9-(Original)

The process of Claim 1 wherein diamond particles are used for providing the roughened surface by abrasion and wherein the diamond particles have a

grain size between about 0.1 to several micrometers, which surface is then cleaned.

-10-(Original)

The process of Claim 1 wherein the pressure on the gas is between about 60 and 240 Torr and at a flow rate of between about 50 and 200 sccm.

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-11-(Original)

The process of Claims 1 or 2 wherein the probe means is elongate and is mounted in the coupler longitudinal axis along the central means chamber means and coupler means with an end of the probe means in spaced relationship to the chamber means; and wherein stage means in the opening of the chamber which forms part of the cavity and provides for mounting the substrate, the stage means having a is in plane around the support surface which a longitudinal axis and which is pre-adjusted towards and away from the plasma in the chamber means so that the substrate can be coated with the diamond film from the plasma.

-12-(Original)

The process of Claims 1 or 2 wherein the insulated chamber is evacuated so that there is less than about 10 ppm of combined oxygen and nitrogen or nitrogen or oxygen alone as the gas which generates the plasma is provided in the chamber.

-13-(Original)

The process of Claims 1 or 2 wherein the substrate is silicon and wherein the substrate holder is molybdenum.

-14-(Original)

The process of any one of Claims 1 or 2 wherein the substrate on which the diamond is deposited has a surface area with a diameter which is greater than about 8 cm.

-15-(Original)

The process of Claim 1 wherein the gas contains about 1% methane.

-16-(Original)

The process of Claim 1 wherein the mode of the plasma is selected from the group consisting of TM012 and TM013.

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-17-(Original)

The process of Claim 1 wherein at pressures of greater than about 250 Torr the stage means can be optionally cooled.

-18-(Original)

The process of Claim 1 wherein the substrate is a silicon carbide seal and the holder is molybdenum which shields a first portion of the seal while allowing a portion of the seal to be coated with the nanocrystalline diamond.

-19-(Previously Presented)

The process of Claim 1 wherein the apparatus has a static magnetic field around the plasma which aids in coupling radiofrequency energy at electron cyclotron resonance and aids in confining ions in the plasma in an electrically insulated chamber means in the coupler means.

REMARKS

Claims 1 to 5 and 8 to 19 are pending. No claims are allowed.

Claim 1 has been amended to correct the antecedent basis errors for purposes of Appeal.

Respectfully,

Ian C. McLeod

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